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VACUUM DIE CASTING OF SILICON  
SHEET FOR PHOTOVOLTAIC APPLICATIONS

First Quarterly Report

For Period Covering

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## ABSTRACT

The objective of this program is to develop a vacuum die-casting process for producing silicon sheet suitable for photovoltaic cells and to develop production techniques for optimization of polycrystalline silicon solar cell output. Efforts will examine process methods which are directed toward minimum cost processing of silicon into a quality suitable for producing solar cells with a terrestrial efficiency greater than 12% and having the potential to be scaled for large quantity production.

In the vacuum die casting technique, silicon is melted under vacuum, and an evacuated die with a thin rectangular cavity is inserted into the melt. Liquid silicon is then injected into the die using a positive pressure of an inert gas. The major portion of the die casting work will be performed at Stanford Research Institute International under subcontract. The initial approach will follow parallel tracks:

- 1) obtain mechanical design parameters by using boron nitride, which has been shown to non-wetting to silicon.
- 2) optimize silicon nitride material composition and coatings by sessile drop experiments.
- 3) test effectiveness of fluoride salt interfacial media with a graphite mold.
- 4) test effect of surface finish using both boron nitride and graphite.

Having established the material and mechanical boundary conditions, a finalized version of the prototype assembly will be constructed and the casting variables determined.

Polycrystalline silicon solar cells, with and without impurities, will be fabricated, characterized, and optimized at ARCO Solar. The major activities will focus on the use of Wacker SILSO, HEM and in-house materials until vacuum die cast wafers are available. A baseline process with vacuum metallized contacts will be established and a reference mass production process with screen-printed metallization and high-throughput diffusions will also be obtained.

## TABLE OF CONTENTS

	PAGE
ABSTRACT	i
TABLE OF CONTENTS	ii
LIST OF FIGURES	iii
I. INTRODUCTION AND SUMMARY	1
II. DIE CASTING OF SILICON SHEET	3
III. MATERIALS CHARACTERIZATION	11
IV. SOLAR CELL PROCESS DEVELOPMENT	15
V. PROGRAM PLAN	21

LIST OF FIGURES

<u>FIGURE</u>	<u>TITLE</u>	<u>PAGE</u>
1.	Representative Thermal Expansion Curves for Various Grades of Commercial Silicon Nitride and Silicon Carbide	4
2.	Plate Die Module For Vacuum Die Casting Sheet Silicon	7
3.	Argon Pressure Injection of Liquid Silicon Into the Die Cavity	8
4.	Pressures Required to Fill	9
5.	Cross-Section of a Boron Nitride Mold Containing A Silicon Rod Produced by Pressure Injection of Liquid Silicon	10
6.	Low Grade Polysilicon	12
7.	"Solar" Grade Polysilicon	13
8.	Reflectance vs. Wavelength	16
9.	Typical Lot Distribution	18
10.	Baseline Process	22
11.	Task I - Vacuum Die Casting Process	23
12.	Task II -Commercial Scale Up 1981-1982	24
13.	Task III - Polycrystalline Silicon Solar Cells	25
14.	Task IV - Economic Analyses	26
15.	Task V - Reporting and Documentation	27

## I. INTRODUCTION AND SUMMARY

This is the first Quarterly Report on JPL Contract 955325: Vacuum Die Casting of Silicon Sheet for Photovoltaic Applications. This program is being carried out by a team consisting of ARCO Solar, Inc. (ASI) as prime contractor, and Stanford Research Institute (SRI) as the major subcontractor.

The program is planned to cover two years. During the first year, the vacuum die casting process for silicon sheet fabrication will be studied, primarily at SRI, and conditions will be determined which will allow the efficient casting of silicon in sheet form, suitable for use in silicon solar cell manufacturing. In parallel with this effort, a study will be made of solar cell manufacturing processes, primarily at ASI, to determine the optimum process sequence for producing cells with conversion efficiencies equal to present cells manufactured from single-crystal silicon, from the polycrystalline material produced by the vacuum die casting process. The major objective of the first year of the program is to achieve the fabrication of 12% efficient solar cells from vacuum die cast material.

If the objective of the first year is achieved, the program will continue into a second year, in which the vacuum die casting and solar cell manufacturing processes will be refined, requirements for scale-up to full production status will be determined, and a detailed cost analysis in the SAMICS format will be prepared. A preliminary cost analysis has been prepared as part of the proposal for this program; assuming a cost of \$10/kg for the polysilicon input material, the vacuum die casting process is anticipated to produce sheet material ready for solar cell manufacture at a cost of approximately 12¢/watt, referred to the final module.

This first quarterly report covers the period from inception of the program (March 16, 1979) through June 30, 1979. As a result of administrative delays, the subcontract with SRI was not finalized until June 21, 1979; therefore only a small amount of experimental work was performed during the reporting period by SRI. Activities at ASI were primarily directed towards establishing a baseline process, to enable detailed comparison to be made between solar cells fabricated on material of widely varying quality, and to developing a surface treatment process which could be applied to polycrystalline material. During the reporting period, the following results were obtained:

\*A detailed program plan covering the first year of the program was negotiated

with JPL. This program plan embodies substantial changes from the original ASI/SRI proposal.

\*At SRI, the first vacuum die cast silicon ingot was produced under this program. This material will be characterized at ASI in the next quarter.

\*A baseline cell process was defined at ASI, and the first production lots of cells, using both standard Czochralski material and polycrystalline material obtained from Wacker, were fabricated and characterized.

\*Reflectivity measurements were made on both single crystal and polycrystalline material texturized by the standard ASI production process, both with and without an additional antireflective coating process step. The results, while incomplete, indicate that an adequately nonreflective surface can be obtained on polycrystalline material by low-cost processing techniques.

\*Several lots of polycrystalline silicon material of widely different properties were obtained for use as starting material for the vacuum die casting process. Chemical analyses of these material lots were made, and Czochralski ingots were grown from them for later comparison with vacuum die cast material.

\*A preliminary study was made of die material requirements and heat transfer requirements for growth of silicon sheet in the vacuum die casting process. On the basis of this study, die materials were ordered and modifications to the existing furnace at SRI designed.

\*Equipment for characterization of small areas of solar cells was specified and ordered by ASI.

During the next quarter, it is anticipated that SRI will achieve fabrication of vacuum die cast ingots having the required dimensions for further processing into solar cells, using input material previously characterized at ASI. ASI will continue work on preliminary etching and surface preparation, and will initiate experimental work aimed at defining optimum diffusion conditions for junction formation in polycrystalline material. It is believed that these two process steps are the only steps in the standard ASI production process which will require major modification in order to fabricate high efficiency cells from polycrystalline material. Input material for the ASI experimental program will be obtained primarily from Wacker and Crystal Systems, since SRI is not expected to be able to produce vacuum die cast material at the rate required by the ASI experimental program during the second quarter.

## II. DIE CASTING OF SILICON SHEET

Die casting is a well known and widely used metallurgical process. The die casting process involves the use of a permanent mold, or die, into which molten metal is forced under pressure. Die casting is inexpensive because permanent molds are used and because heat transfer rates are high, resulting in high machine throughput. Die castings have excellent dimensional control, surface finish and mechanical properties.

Typically, die castings are made from aluminum-silicon or zinc alloys, using steel molds. In order to apply the die casting process to the casting of silicon sheet, a suitable mold material must be found. The primary requirements for the mold material are that it not be attacked by molten silicon, and that it have a coefficient of thermal expansion lower than silicon since silicon expands on freezing.

No material is known which is completely nonreactive with molten silicon. However, the requirements for a die casting mold material are easier to meet than the critical materials requirements in most other silicon sheet processes. In the die casting process, the mold material is in contact with molten silicon for a very short time during each casting cycle, because of the rapid solidification characteristic of the die casting process. A number of materials are known which are very slowly attacked by molten silicon and which do not grossly contaminate the silicon. Examples are silicon dioxide (fused quartz), which is used as a crucible material in the Czochralski process, high purity graphite and silicon carbide, silicon nitride and silicon oxynitride. All of these materials have the required mechanical properties at the casting temperature to maintain dimensional stability.

A low coefficient of thermal expansion is required so that the mold will not exert any constraint on the silicon while the silicon is cooling from the solidification temperature. In order to prevent any constraint during cooling, the mold material must have a lower thermal expansion than the silicon casting, and some degree of taper may be necessary in the mold cavity. As shown in Figure 1, all of the above listed materials have a thermal expansion coefficient lower than silicon in the

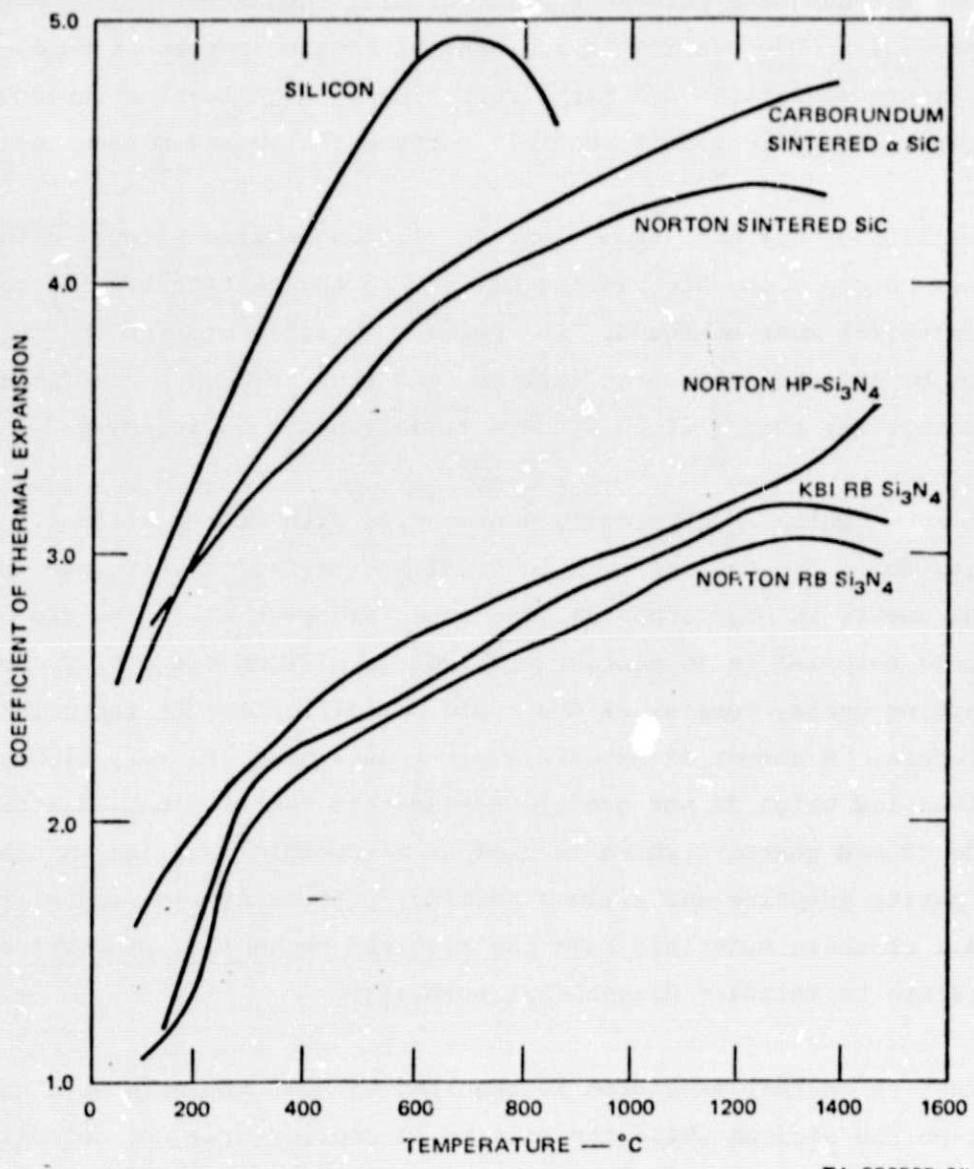


Figure 1. REPRESENTATIVE THERMAL EXPANSION CURVES FOR SILICON AND VARIOUS GRADES OF COMMERCIAL SILICON NITRIDE AND SILICON CARBIDE

temperature range of interest. Fused quartz, which is not shown on Figure 1, has a lower thermal expansion than any of the materials shown.

The required mold parts can be most readily and economically fabricated in reaction-bonded silicon nitride or silicon oxynitride and sintered silicon carbide. Techniques such as warm molding and isostatic pressing are routinely used to produce complex parts of high-dimensional accuracy. It is possible, but more expensive, to make the molds from hot pressed silicon nitride, using diamond grinding. It is least feasible to fabricate the required parts in fused silica, because of the difficulty of obtaining the necessary high tolerances.

The above considerations suggest reaction-bonded silicon nitride and sintered silicon carbide as the most suitable mold materials for the vacuum casting of silicon. The extent of the reaction between liquid silicon and pure dense silicon nitride, silicon oxynitride or silicon carbide has not been investigated extensively. Results cited in the literature suggest that silicon nitride and silicon oxynitride undergo minimal reaction with liquid silicon and should be excellent containment materials. Silicon carbide is probably also suitable although incorporation of silicon carbide into the silicon has been shown to lower solar cell conversion efficiency.

Reaction-bonded silicon nitride can contain up to 20% pores, which might be infiltrated by molten silicon, thereby promoting adherence between the wafer and the mold on freezing. The extent of this effect depends on the macro- and micro-wetting characteristics of the particular silicon nitride/silicon interface and will be influenced by the surface finish of the mold and by the size and number of pores. These are controllable variables, which we propose to investigate. If surface porosity is a limiting feature of reaction-bonded silicon nitride, surfaces will be coated with a dense layer of CVD silicon nitride or silicon oxynitride.

Sintered silicon carbide has a much lower porosity than reaction-bonded silicon nitride, but has not been evaluated specifically for reaction with liquid silicon. Hot pressed silicon carbide does react slightly, whereas reaction between dense CVD silicon carbide and silicon might be insignificant. Thus sintered silicon carbide, with and without a CVD coating, will be an alternative die material to be evaluated in this program.

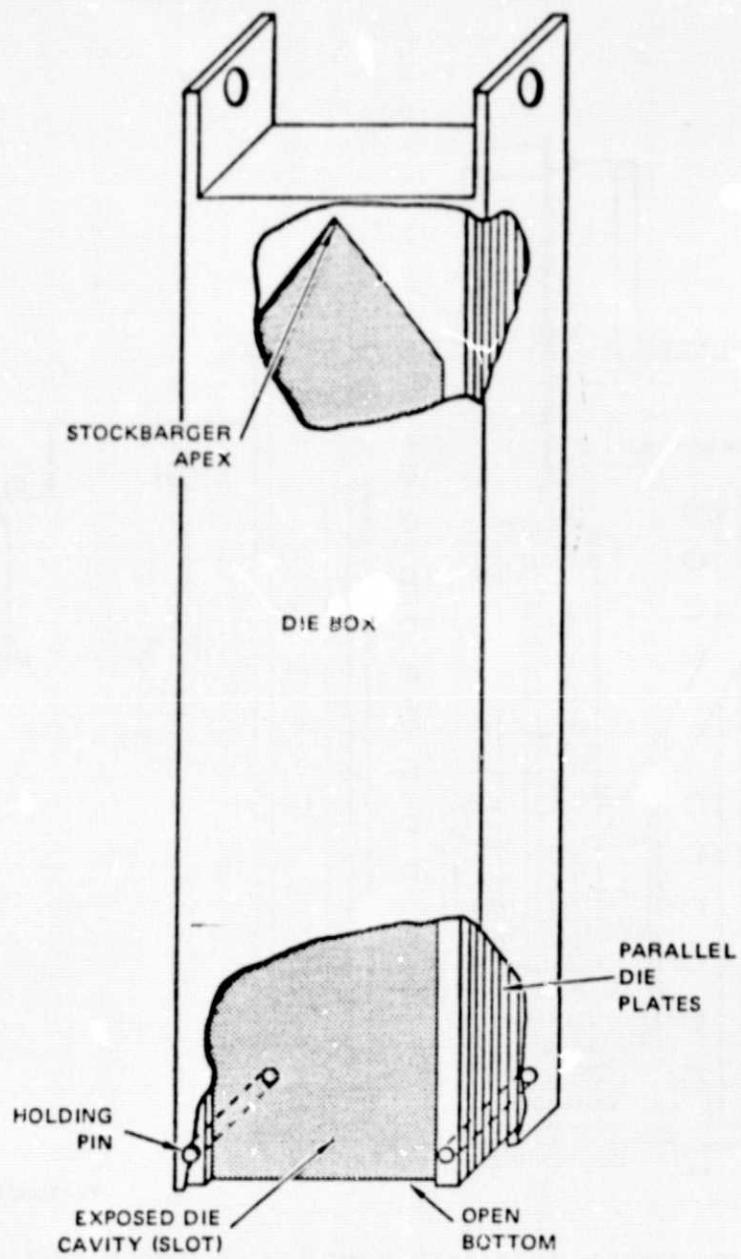
The experimental work to be carried out during the first year of the program will utilize a modified Czochralski crystal grower. Silicon sheets will ultimately be cast using a multiple sheet die, as shown in Figure 2. The individual die plates will be formed so the mold cavities will produce silicon sheets of approximately 0.012 inches in thickness. The width of the sheets will be limited by the crucible diameter, and the maximum length is limited by the available pressure.

The experimental apparatus is shown in Figure 3. After melting the silicon in the crucible, the apparatus is evacuated by the vacuum pump. The lower end of the mold is then inserted into the pool of molten silicon, and argon gas is injected into the apparatus to force the molten silicon into the mold. Heaters and heat shields are used so that the mold is preheated to a temperature somewhat below the melting point of silicon, with a longitudinal temperature gradient to provide directional solidification. Crystallization is initiated at the apex of the mold and proceeds downwards. The mold is withdrawn from the molten silicon before crystallization is entirely complete; the remaining molten silicon in the mold is retained by surface tension and gas pressure.

The pressure required to force molten silicon into the mold is considerably dependent on the slot width, as shown in Figure 4. Because the surface tension of molten silicon is so high, it should be possible to control the gas pressure to fill a long mold, without exerting so much pressure that molten silicon is forced into the small gaps between the edges of the die plates.

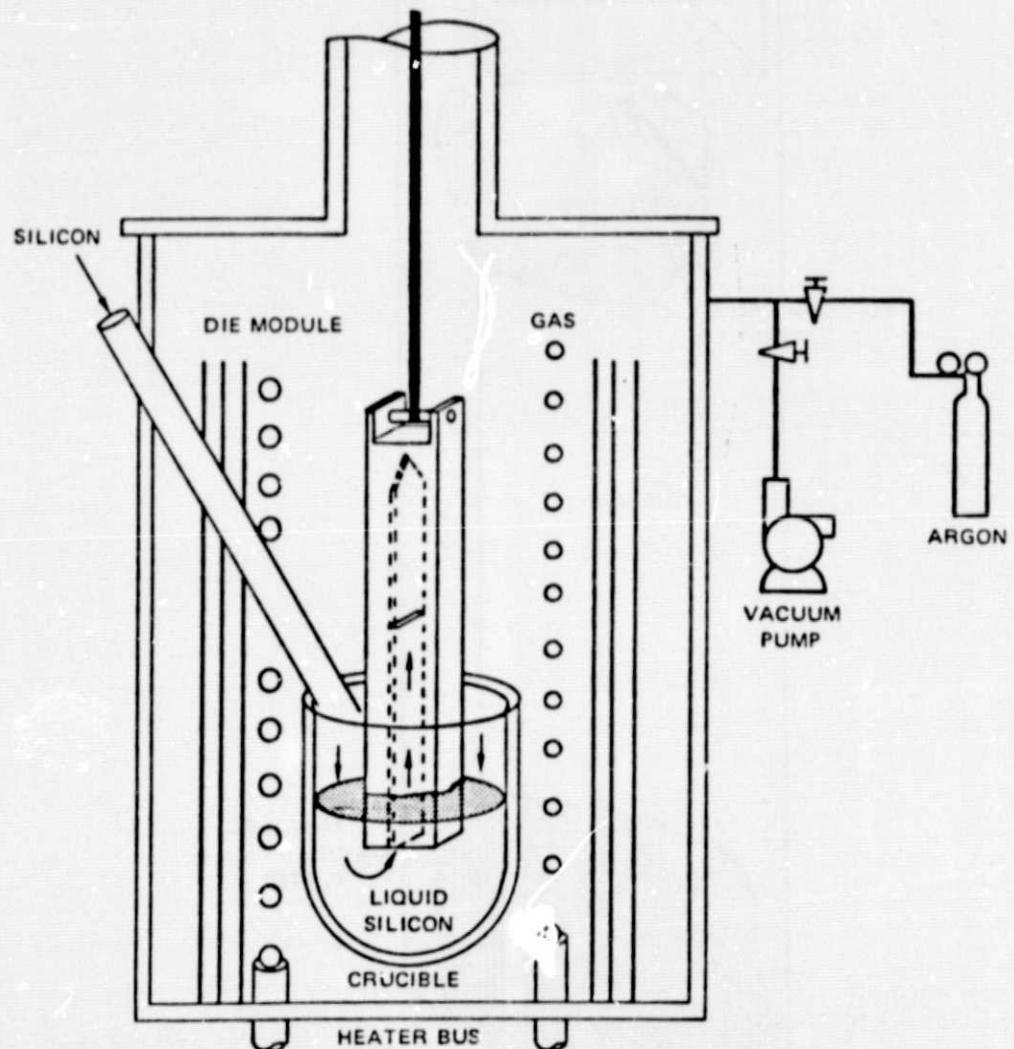
During the first quarter of the program, a silicon ingot was successfully cast in an experimental apparatus at SRI. A photograph of this ingot is shown in Figure 5. The ingot was cast from a melt of semiconductor-grade polysilicon into a mold cavity machine into hot-pressed boron nitride. The dimensions of the ingot were 3 inches long x 1/8 diameter. This experiment was performed merely to verify the readiness of the experimental apparatus to undertake the work planned for this program; the results show that the furnace, vacuum system and argon injection system are working properly.

High-density graphite and several varieties of silicon nitride have been ordered for fabrication of experimental molds. The experimental furnace will take an 8" diameter crucible, and experimental molds will be fabricated up to 24" in length. Molybdenum heat shields have been designed and are being fabricated to provide the proper longitudinal temperature gradient for directional solidification.



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Figure 2. PLATE DIE MODULE FOR VACUUM DIE CASTING SHEET SILICON



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Figure 3. ARGON PRESSURE INJECTION OF LIQUID SILICON INTO THE DIE CAVITY

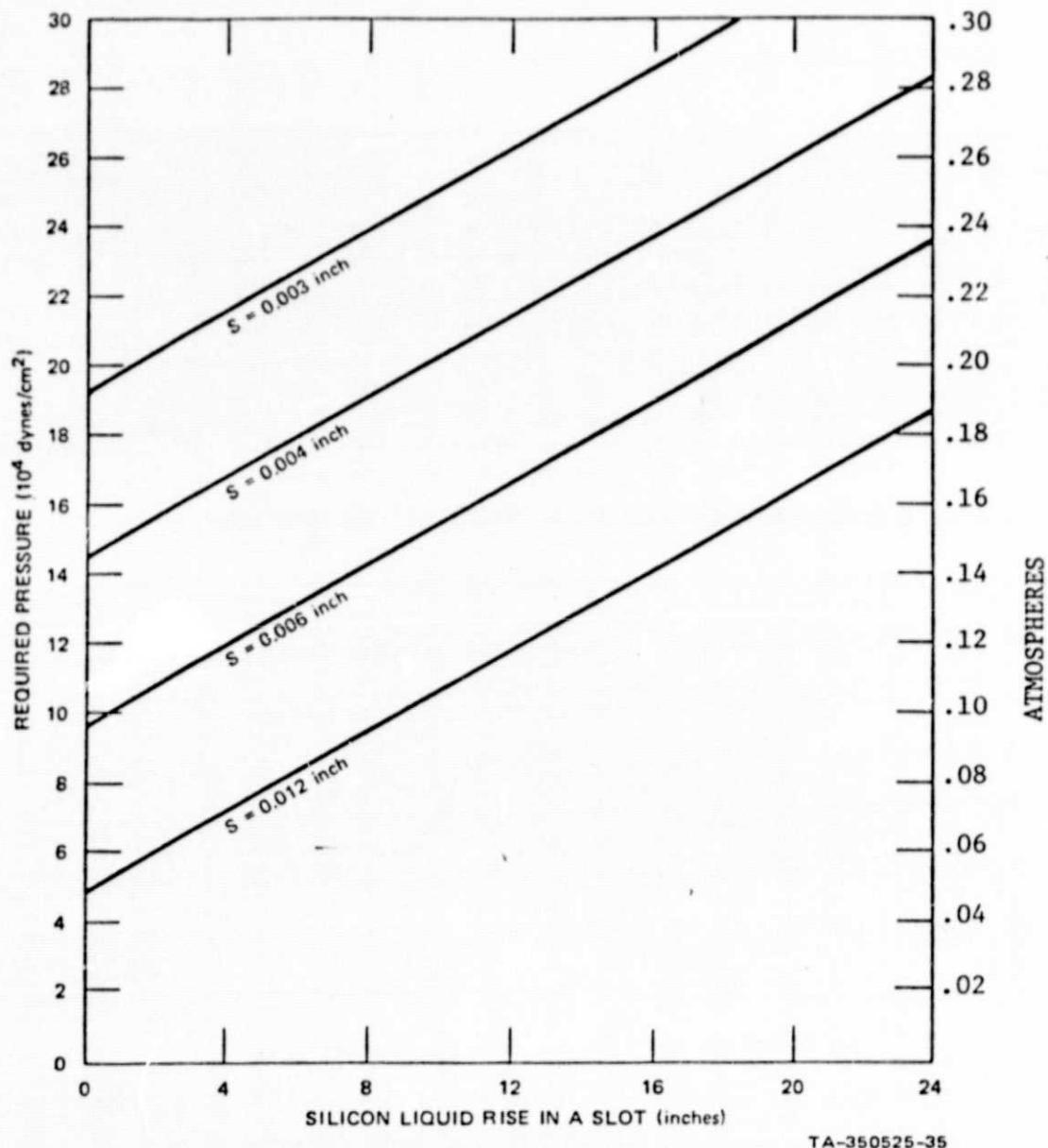
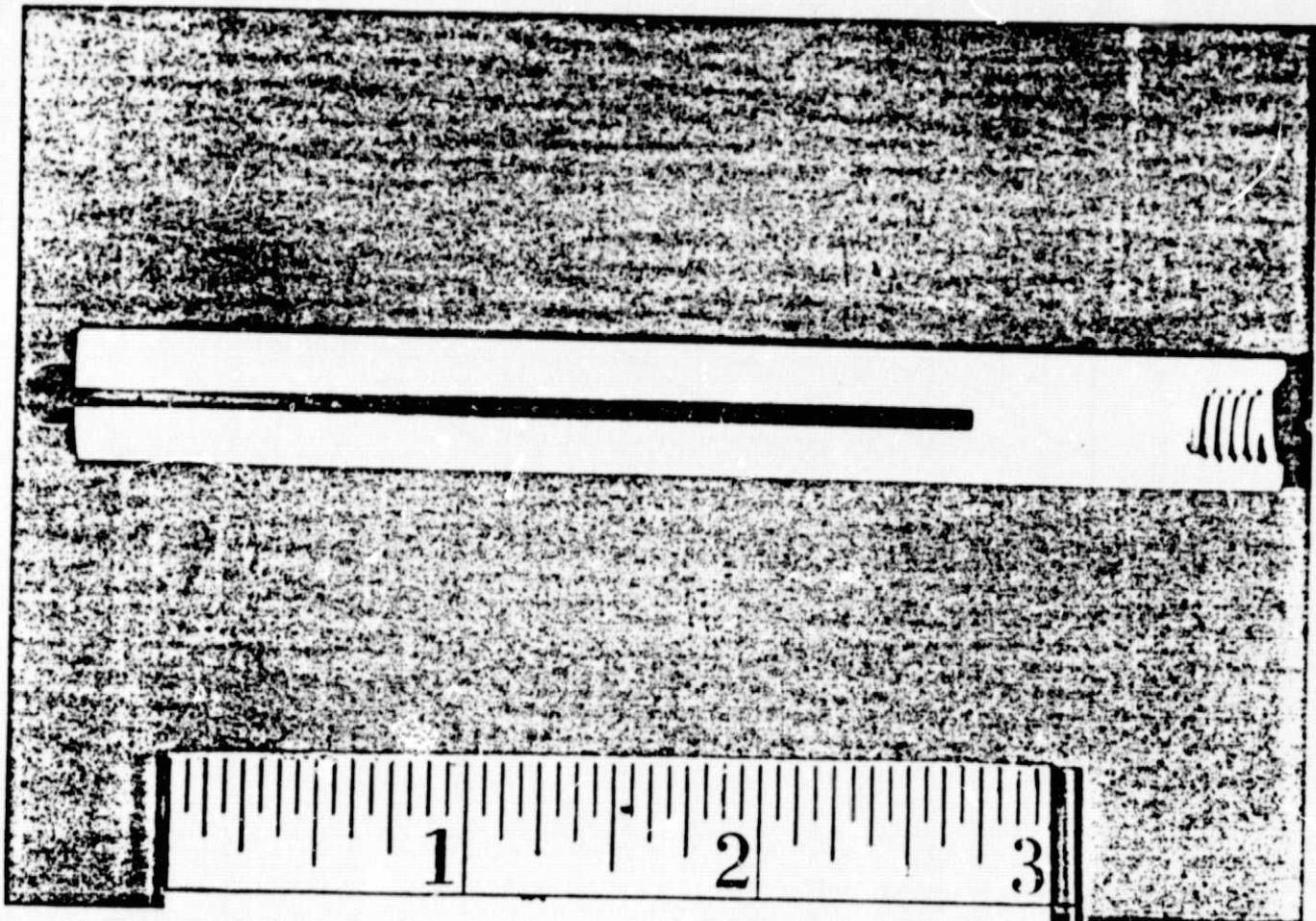


Figure 4. PRESSURES REQUIRED TO FILL



CROSS-SECTION OF A BORON NITRIDE MOLD CONTAINING A  
SILICON ROD PRODUCED BY PRESSURE INJECTION OF LIQUID SILICON

Figure 5.

### III. MATERIALS CHARACTERIZATION

ASI has procured two lots of polysilicon of lower quality than standard semiconductor-grade polysilicon. This material will be utilized in the program to evaluate the effects of lower quality starting material.

It is evident that the silicon sheet produced by the vacuum die casting process will contain many more structural imperfections than the present Czochralski grown single-crystal material used for solar cell manufacture. It is also likely to contain more chemical impurities resulting from reaction of the molten silicon with the mold material and the use of lower cost polysilicon. While the cooling rate during solidification, and hence the average grain size in the die cast sheet, can be controlled to some extent by control of the temperature environment of the mold during the casting process, a trade-off will probably be found to exist between structural imperfections and chemical contamination.

ASI has therefore planned to evaluate starting material of various degrees of purity in the standard Czochralski process. The same starting materials will then be used by SRI in the vacuum die casting apparatus in order to enable ASI to make a direct comparison between solar cells made from equivalent starting material. This comparison should assist us in determining the nature of the material imperfections characteristic of die cast material and their effects upon device yield.

The first lot of lower quality material is "burn-out" material. This is material produced during the first run of a polysilicon reactor after cleaning, and is contaminated with a variety of residual materials from the cleaning process. "Burn-out" material is normally either discarded or recycled for trichlorosilane production. A spectrographic analysis of this material is given in Figure 6.

The second lot of lower quality material is polysilicon produced by the reduction of silicon with carbon, using high purity starting materials; it is therefore essentially a premium grade of metallurgical silicon. A spectrographic analysis of this material (labeled "solar grade silicon") is shown in Figure 7.

Czochralski ingots were grown from both lots of material at ASI. Both melts were "dirty" and so both ingots were polycrystalline. Solar cells will be made from both ingots, and the results will be included in the next report.

FIGURE 6  
LOW GRADE POLYSILICON

Boron	1 PPM
Aluminum	1 PPM
Iron	2 PPM
Chromium	70 PPM
Nickel	2 PPM
Manganese	2 PPM
Copper	3 PPM
Titanium	80 PPM
Vanadium	1 PPM
Zirconium	2 PPM
Cadmium	2 PPM
Barium	70 PPM
Magnesium	2 PPM
Sodium	50 PPM

FIGURE 7  
"SOLAR" GRADE POLYSILICON

Boron	1 PPM
Aluminum	25 PPM
Iron	20 PPM
Chromium	50 PPM
Nickel	1 PPM
Manganese	1 PPM
Copper	1 PPM
Titanium	1 PPM
Vanadium	1 PPM
Zirconium	1 PPM
Cadmium	5 PPM
Barium	50 PPM
Magnesium	1 PPM
Sodium	50 PPM

When SRI has determined the mold materials which they will use for the bulk of their experimental runs, Czochralski ingots of high purity material deliberately contaminated with mold material will be grown at ASI for comparison with die cast sheet material.

#### IV. SOLAR CELL PROCESS DEVELOPMENT

ASI's primary task during the first year of this program is the development of a solar cell process sequence which will yield cells with characteristics comparable to present standard production cells, starting from imperfect polycrystalline sheet material. Previous work at ASI and elsewhere has shown that solar cells of reasonable conversion efficiency can be produced from large grain polycrystalline wafers, and also from single-crystal wafers of considerably lower purity than present industry standards; therefore present process sequences are clearly adaptable to material of lower perfection and purity than is presently used. We anticipate that the vacuum die casting process, although it will provide material of still lower perfection than any material we have previously worked with, should at least provide material with reproducible characteristics. It therefore appears to be feasible to determine an optimum process sequence for this material during the first year of the program. The process modifications found to be useful for die cast sheet material should also be useful in processing imperfect material produced by other processes.

We anticipate that major modifications will be required in starting wafer specifications, surface preparation and junction formation steps. It may also be desirable to add annealing or gettering steps to the cell process sequence.

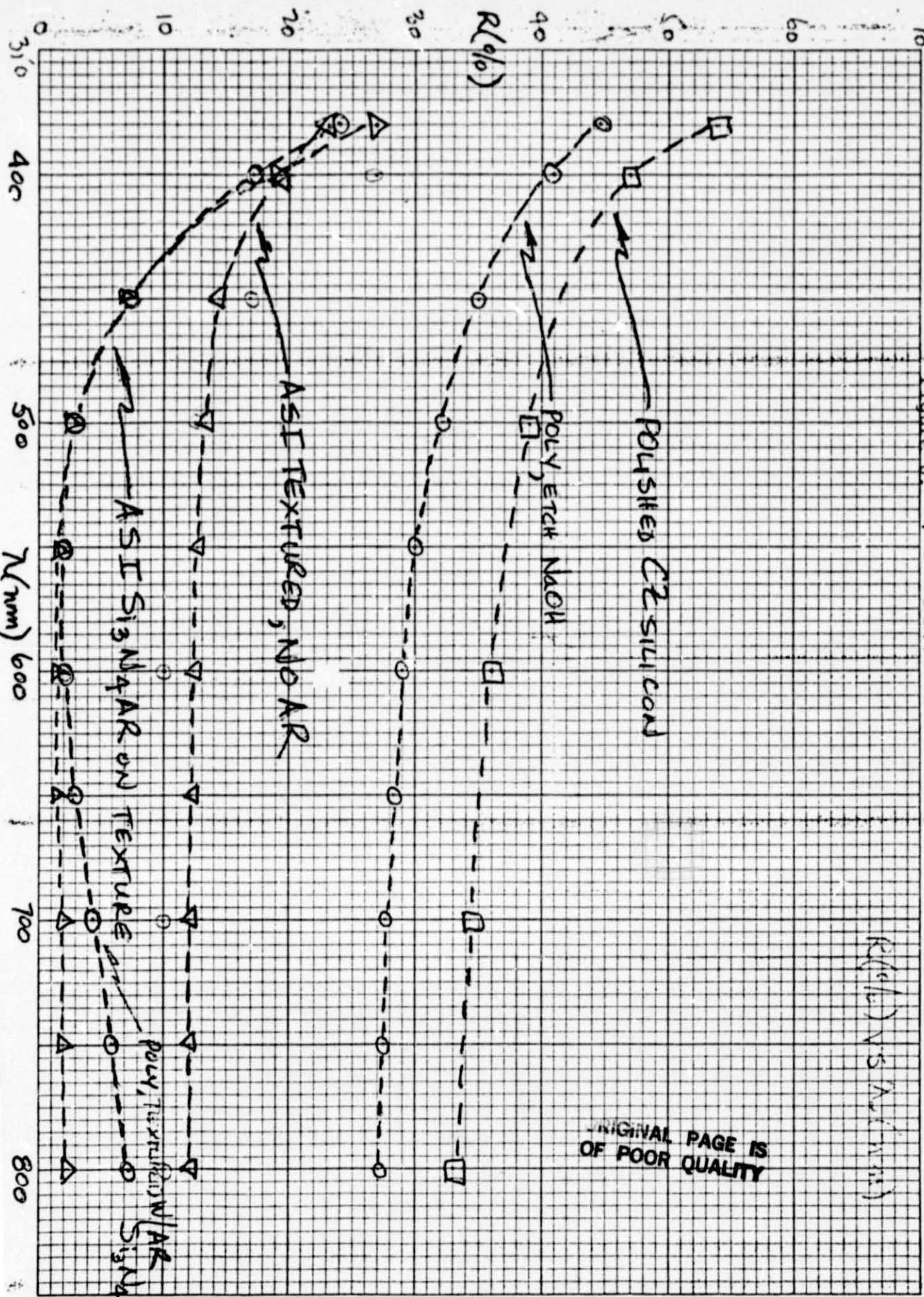
During the first quarter of this program, attention was focused on the surface preparation step. The standard ASI production process utilizes a two-step etching process, in sodium hydroxide solutions of approximately 20% and 2% sodium hydroxide concentration, respectively. The first step removes surface damage and contamination, and the second step produces a "textured" surface on the (100)-oriented wafer surface. The surface texture provides effective suppression of optical reflection when the cell is encapsulated, without the necessity for an additional anti-reflective coating.

It would be anticipated that this surface preparation process would not produce a properly textured surface on polycrystalline material with randomly oriented grains. Optical reflection measurements performed on both standard and polycrystalline material show that this is indeed the case. The results of these measurements are summarized in Figure 8. The polycrystalline material used was Wacker Silso<sup>TM</sup>, a large grain polycrystalline material produced by a casting process.

Figure 8. REFLECTANCE VS. WAVELENGTH

$R(\text{nm}) \times 10^{-3} \text{ (arbitrary)}$

ORIGINAL PAGE IS  
OF POOR QUALITY



The first three curves in Figure 8 show optical reflectance vs. wavelength for a polished monocrystalline silicon wafer, a sodium hydroxide textured polycrystalline wafer, and a sodium hydroxide textured monocrystalline wafer of (100) orientation, respectively. At 600 nm, the reflectances are 0.36, 0.29, and 0.125, respectively. The reflectance of the polished surface is in agreement with literature values. The textured monocrystalline surface has a reflectance approximately equal to the square of the reflectance of the polished surface, showing that the texturing process has been effective in producing a surface which reflects light through two reflections. After encapsulation in polyvinyl butyral, the monocrystalline surface can be calculated to have a reflectance of approximately 0.04. The reflectance of the polycrystalline sample is nearly as great as that for a polished surface, indicating that the texturing has not been effective in producing a "two-bounce" surface because of the random orientation of the grains. After encapsulation, the polycrystalline sample can be calculated to have a reflectance of 0.10. This is high enough so that consideration of the addition of an anti-reflective coating step to the process is justified.

The bottom two curves show the reflectance of the textured polycrystalline sample and the textured monocrystalline sample, after deposition (CVD) of a silicon nitride AR coating. The reflectance of both samples has been reduced to a level which will result in a negligible performance penalty after encapsulation, although the polycrystalline sample is still noticeably higher in reflectance.

We can conclude that, for polycrystalline material, the standard process sequence should be modified to delete the texture etch in 2% sodium hydroxide, since it is not sufficiently effective in reducing reflectance, and to add a low cost AR coating step after contact formation. Potential candidates for the additional process step are evaporation, CVD deposition, and spin-on or dip coatings.

Figure 9 summarizes results of an experimental lot of polycrystalline wafers (Wacker Silso<sup>TM</sup>) processed into solar cells using the standard ASI production process, including screen printed contacts. The I-V curves are tightly grouped, showing that the material is capable of providing as reproducible results as standard monocrystalline material. In comparison to standard ASI production cells, the following differences can be noted:

Figure 9.

TYPICAL LOT DISTRIBUTION

WACKER SILSO  
\* PASTE CONTACTS  
\* NO AR COATING

CURRENT, A

3.0

2.0

1.0

0.1

0.2

0.3

0.4

0.5

0.6

VOLTAGE, V

OF POOR QUALITY

(1). The short circuit current density is significantly lower, averaging  $21.5 \text{ mA/cm}^2$  while standard ASI production lots would average  $27 \text{ mA/cm}^2$  when measured in air, before encapsulation. However, this difference is almost entirely accounted for by the difference in optical reflectance; there is no evidence that the grain boundary recombination in the polycrystalline cells leads to an important loss in current output.

(2). The open circuit voltage is slightly lower, 0.54 V compared to 0.58 V for monocrystalline cells, and there is a noticeable shunt conductance in the polycrystalline cells. These differences can be accounted for as effects of grain boundary leakage currents. Presumably fine grain polycrystalline material, as produced by die casting, will show larger shunt conductance and open circuit voltage loss. This indicates the need for modification of the junction formation process step to minimize the effect of grain boundaries on junction leakage.

(3). The curve factor and apparent series resistance of the polycrystalline cells are comparable to standard ASI production cells. This indicates that the standard ASI contact process can be applied to polycrystalline cells without significant modification.

During the next quarter of the program, attention will be focused on the junction formation process step. Diffusion parameters will be varied in an attempt to minimize junction leakage currents at grain boundaries. One approach that will be tried is the use of a two step phosphorus diffusion as suggested by Di Stefano and Cuomo of IBM. A similar diffused region impurity profile can be obtained by a single step diffusion using a spin-on diffusion source containing both arsenic and phosphorus, and this approach will also be evaluated.

It is not anticipated that the die casting work at SRI will be sufficiently advanced during the second quarter to provide sufficient material for the cell process development work. In addition to the Wacker material, polycrystalline material produced by the heat exchanger method at Crystal Systems, Inc., is on order and should be received in time for initial evaluation.

Although cell process sequences will be modified continuously during the program, it is desirable to define a baseline process sequence which can be used as a reference point for comparisons of materials and process modifications. A baseline process has been defined for this program, and is presented in Figure 10. It is known to provide cells with characteristics substantially identical to the ASI production process, and is easy to carry out in the laboratory.

Testing of polycrystalline wafers and cells has indicated that characterization of small areas is an essential element of a complete test program. ASI has under development a laser scanner which will provide detailed information on the photo-response characteristics of elemental areas of solar cells, but this instrument is not expected to be available during the second quarter of the program. Until it is ready, reliance will be placed on scanning electron microscope observations in the electron beam induced current mode to locate areas of low photoresponse in cells, and on spreading resistance probe measurements to characterize variations in electrical properties of wafers.

## V. PROGRAM PLAN

The plan for the program was extensively revised during the first quarter. The revised program plan is presented on the charts following this section. While the entire program is planned as a two year effort, only the first year is shown on the charts. Effort during the second year is dependent on the achievement of the key milestone of fabrication of a 12% efficient cell from vacuum die cast material by the end of the fourth quarter of effort.

The program plan is broken down into 5 major tasks:

1. Die Casting Process Development
2. Die-Casting Scale-Up
3. Polycrystalline Cell Process Optimization
4. Economic Analysis
5. Reporting and Documentation

Effort on task 2 will occur primarily during the second year of the program; during the first year, the ultimate objective of scaling up the experimental die casting process to production levels will be kept in mind, but it would be inappropriate to develop detailed plans for a production die casting facility until the process has been thoroughly characterized and proven in the laboratory. Task 4 involves preparation of a SAMICS/SAMIS analysis of the complete process. Because of the low level of effort on task 2 during the first year, the SAMICS/SAMIS input for the die casting process is expected to be preliminary in nature. The information of cell process optimization resulting from the effort on task 3 during the first year of the program is also expected to be preliminary, but because the anticipated process sequence involves only modifications of an established production process the available information, at the end of the first year, should be sufficient for a detailed analysis.

Figure 10. Baseline process

1. ETCH - NaOH 20%, 2%
2. CLEAN - acid
3. DRY
4. DIFFUSE -  $\text{POCl}_3$  - 0.3  $\mu\text{m}$  junction
5. BACKETCH -  $\text{HNO}_3$ -HF-  $\text{HOAc}_C$
6. REMOVE  $\text{P}_2\text{O}_5$  - HF
7. CONTACT BACK - Ti-Pd-AG (1500 $\text{\AA}$ -500 $\text{\AA}$ -3  $\mu\text{m}$ )
8. CONTACT FRONT- Ti-Pd-Ag - 6 grids/2cm x 2cm
9. EDGE ETCH - plasma
- 10 TEST AND CHARACTERIZE

ARCO Solar, Inc. ♦ PLANNING SCHEDULE	TITLE	TASK I-Vacuum Die Casting Process	ACCOUNT NO.	DATE	7/9/79											
					PREPARED BY			APPROVED			REV.			Feb. 80		
	Mar	Apr	May	June	July	Aug	Sept	Oct	Nov	Dec	Jan	Feb				
prepare vacuum furnace																
order and test die materials																
define die geometry																
final material selection																
fabricate and cast Large sheets																
define casting variables																
replicate castings																



ARCO Solar, Inc.	PLANNING SCHEDULE	TITLE	TASK-III Polycrystalline Silicon Solar										ACCOUNT NO	DATE	7/9/79		
			PREPARED BY														
			Mar	Apr	May	June	July	Aug	Sept	Oct	Nov	Dec	Jan	Feb	FEB	30	TOTAL
List proposed sheet																	
Procure sheet																	
Characterize Sheet																	
Estab. baseline process																	
Fab. & char. baseline cells																	
Modify process & evaluate																	
Cells in die-cast sheet																	



Vacuum Die Casting